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CARBOXYMETHYLCELLULOSE FROM COTTON LINTERS

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Production of carboxymethylcellulose (CMC) from wood pulp shows a steady increase. Unfortunately, the CMC produced from wood pulp does not have a high viscosity (1). It has been reported earlier (2, 3) that high-molecular-weight CMC can be obtained from purified cotton linters.

The authors have developed technology for the preparation of high-viscosity CMC from untreated cellulose, that is, from raw linters--an intermediate of the linters industry.

To produce CMC with the desired properties, they have studied the etherification of raw linters with sodium monochloroacetate. Raw linters have the following composition: cellulose 80-85%, wax and fat 1-1.5%, pentosans 2.5-3.5%, ash 2-2.5%, lignin 1.5-2.5%, foreign matter 1.5-2.6%.

The etherification reaction was studied as a function of the mole ratio of the reactants, temperature and reaction time. It was established that at a specific temperature and mole ratio the degree of substitution (DS), in terms of the carboxymethyl content of the finished product, amounts to 25-85% (Fig. 1). At 40°C, the DS is about 60-65%. With increase in temperature of the reaction mixture to 80°C, the DS increases to 85-90% (Fig. 2).

Under these conditions, CMC was prepared from raw linters by means of a modified experimental apparatus (Fig. 3). Air-dry raw linters (type IV, grade II) in bales (1), opened up in apparatus (2), were charged to "crusher-mixer" (3), with the addition of a calculated amount of NaOH solution. When the mass became homogenous, the etherifying agent (sodium monochloroacetate) was added and the reaction mass mixed for 90 additional minutes. The reaction mixture was ripened 30 minutes

in reservoir (4). The product was dried in sections (7, 8, 9) and subsequently analyzed (GOST 5588-70).

This technology was used for obtaining CMC from cotton linters at the Vladimir chemical plant, at the Engels chemical fibers plant, at the pilot plant of the Vladimir Scientific Research Institute for Synthetic Resins (VNIISS) and at the Scientific Research Institute of Chemistry and Technology (NIIKHTTS). The finished product was compared with CMC obtained from raw linters.

The cellulose from the Engels and Vladimir plants was compact, but the sample received from the NIIKHTTS pilot plant, after treatment, was loose and fluffy (Table 1). The physicochemical properties attributable to the various forms of cellulose differ considerably. CMC of high viscosity was obtained from the raw linters of the Alimkent plant. CMC produced from raw linters is slightly less soluble than the commercial products.

The technology described above does not include mercerization, because during beating of raw cotton and mixing it with the reagents, a calculated amount of sodium hydroxide solution was added, thereby shortening the time required for breaking up the alkali cellulose to 60 minutes. This procedure has only a slight effect on the etherification of alkali cellulose and, consequently, on the water solubility of the finished product.

The process for preparation of CMC from raw linters was based on the considerations mentioned earlier (Table 2).

Table 2
Conditions for Production of CMC
from Purified and Raw Cotton Linters

Characteristics	Cotton Linters	
	Purified*	Raw**
Mercerization temperature, °C	26	--
Duration of mercerization	60	--
Removal of excess alkali from alkali cellulose	2.6	--
Alkali content of alkali cellulose, %	13	17
Duration of beating of alkali cellulose, min.	120	90
Temperature of reaction mixture during beating, °C	27	15-20
Etherification temperature, °C	23-35	23-35
Mixing time of alkali cellulose with sodium monochloroacetate, min.	120	90
Ripening temperature of the etherification process, °C	85-90	85-90

* Purified cotton linters (pressed)
** Fluffy raw linters

The technology for producing CMC from raw linters with the modified apparatus did not include the preparation of alkali cellulose to save the cost of development work. The product differs considerably in physicochemical properties from the known types of CMC (Table 3).

The laboratory values were confirmed with the experimental samples of CMC prepared from raw linters at the Namangan plant.

The high-viscosity CMC prepared from raw linters has satisfactory physicohemical properties and good water solubility. It may be used as stabilizer of drilling muds in oil and gas wells and also to stabilize soil and dunes or to protect against erosion in the building and farming industries.

Table 3
Physicochemical Characteristics of CMC
Produced from Sulfate Pulp, Purified and Raw Linters

Characteristics	From Sulfate Pulp*	From Purified Cotton Linters	From Raw Cotton Linters
Degree of polymerization	580	850	1200
Degree of substitution according to carboxymethyl content, %	82	72	80
Content of basic substance, %	50	50	52
Viscosity of 0.5% aqueous solution, cps	15	22	28-30
Solubility in water, %	99.6	99.6	99.1
NaOH content, %	22	21	20
Alkali content of product, %	1.1	0.98	--
pH	7.0	7.3	7.0

Note: *Trademark CMC 85/600,, manufactured at the Namangan chemical plant, according to GOST 588-70.

Table 1
CMC Obtained from Various Types of Cotton Linters

Sample	Cellulose			CMC				
	Content %	DP %	Ash %	DP %	DS %	Viscosity of 0.5% Aqueous Solution	Content of Basic Substance %	Solubility in Water %
Vladimir plant	99.3	2150	0.16	850	82.0	23.5	52	99.5
Engels chemical fibers plant	99.1	1800	0.07	750	83.0	20.2	51	99.3
NIIKHTTS pilot plant	92.2	1180	0.20	670	81.5	18.1	51	99.6
Short-fibered raw linters from Alimkent plant								
BLO-6 linters, grade 3	82.3	2900	2.50	1080	80.5	28.2	52.5	90.2
BLO-6 linters, grade 4	80.9	2620	2.70	980	82.5	25.1	53.1	99.1

Note: Mole ratio of cellulose:sodium hydroxide:sodium monochloroacetate = 1:1.9:1.85
Apparatus of BLO type

Figure 1
DS of CMC as a Function of Time Required to Consume 1, 2 and 3 Moles of Sodium Monochloroacetate

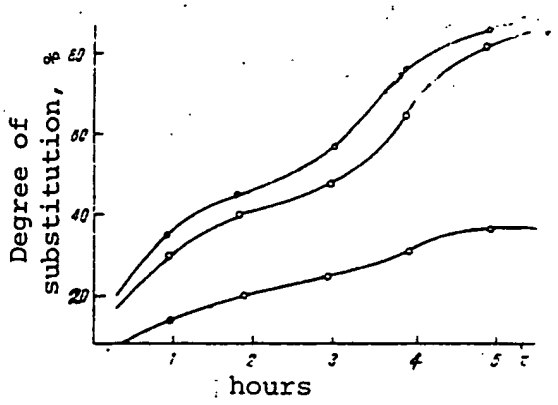


Figure 2
DS of CMC as a Function of Consumption of Moles of Sodium Monochloroacetate per Mole of Alkali Cellulose at 40°C (Curve 1) and at 80°C (Curve 2)

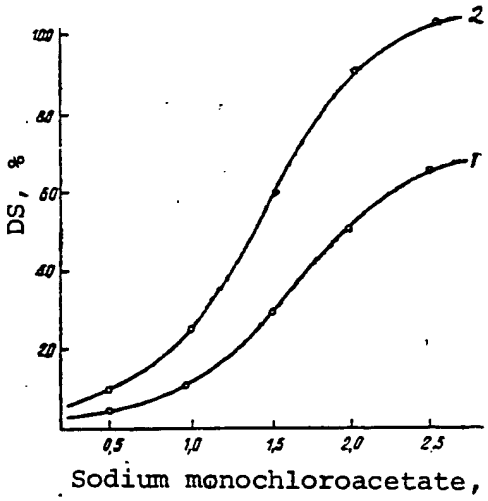
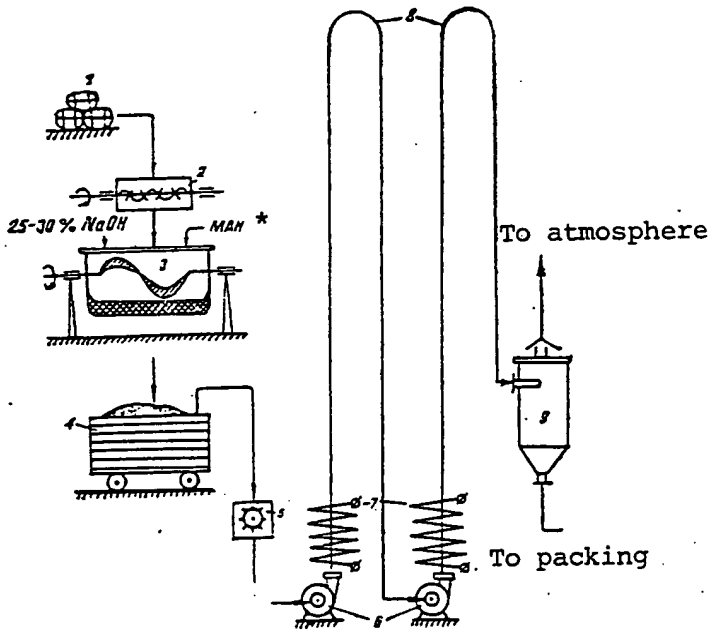


Figure 3
Schematic Diagram for Production of CMC
from Cotton Linters

1 - cotton linters; 2 - opener; 3 - beater-mixer;
4 - ripening reservoir; 5 - opener; 6 - fans;
7 - heater; 8 - dryer; 9 - cyclone
*sodium monochloroacetate



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